Data Validation Report

Project #69420

Peoples Gas-Willow Street/Hawthorn Avenue

Water and Soil Vapor Sample Analyses Performed by

Pace Analytical Services, Green Bay and TestAmerica, Inc., Pittsburgh and STAT Analysis Corporation Chicago, IL

Prepared for



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February 10, 2014

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1.0 INTRODUCTION

All data validation was performed by Shepherd Technical Services following US EPA National Functional Guidelines (NFG), where applicable, using electronic deliverables. Guidance and requirements appearing in the NRT Multi-Site Quality Assurance Project Plan, Rev. 2, 2007 ("Multi-Site QAPP") were also used in the validation process.

STAT Analysis Corporation performed the sample analyses on the soil vapor samples. The laboratory maintains accreditation under the Illinois EPA Environmental Laboratory Accreditation Program (IEPA ELAP #100445). The laboratory is also accredited under the National Environmental Laboratory Accreditation Program (NELAP) by the Oregon Environmental Laboratory Accreditation Program (ORELAP #IL300001).

Pace Analytical Services, Inc., Green Bay, WI performed the analyses for metals, PAHs, and PVOCs on the groundwater samples. The Pace Green Bay laboratory maintains certification under the Illinois EPA Laboratory Accreditation Program (ID #200050). The Pace laboratory is also accredited under the National Environmental Laboratory Accreditation Program (NELAP) by the Florida Department of Health Environmental Laboratory Certification Program (ID #E87948).

Analyses for Available Cyanide were performed by the TestAmerica Pittsburgh laboratory. TestAmerica Pittsburgh maintains accreditation under the National Environmental Laboratory Accreditation Program (NELAP) by the Pennsylvania Department of Environmental Protection Laboratory Accreditation Program (Certificate Number 008, ID#2-00416) as well as the Illinois EPA Environmental Laboratory Accreditation Program (IEPA ELAP #200005).

The laboratories provided all analytical data, including all internal laboratory QC results in an electronic deliverable format to facilitate the validation process.

A total of 24 aqueous samples including 4 field blanks and 14 soil vapor samples were collected December 17, 2013 to December 20, 2013 at the Peoples Gas-Willow Street/Hawthorne Avenue sites. Samples were organized into 18 sample delivery groups (SDGs, or laboratory lot numbers). Samples were organized into four sample delivery groups (SDG or laboratory lot number) for the analyses by the PACE laboratory and nine SDGs by the

TestAmerica laboratory and five SDGs by STAT laboratory. Samples were analyzed for the indicated parameters using the methods listed in Table 1-1

Table 1-1. Sample/SDG Cross Reference

Matrix	Field ID	STAT/PACE Sample ID	TestAmerica Sample ID	EPA 3C	EPA TO- 15	EPA 6020	EPA 8260	EPA 8270 by SIM	OIA- 1677
Air	121813007	13120429-001		Х	Х				
	121813008	13120429-002		Χ	Х				
	121813011	13120431-001		Χ	Х				
	121813014	13120429-003		Χ	Х				
	121813016	13120430-001		Χ	Х				
	121913019	13120466-001		Χ	Х				
	121913021	13120466-002		Χ	Х				
	121913022	13120466-003		Χ	Х				
	121913024	13120466-004		Χ	Х				
	121913025	13120466-005		Χ	Х				
	121913026	13120466-006		Χ	Х				
	121913028	13120467-003		Х	Х				
	121913030	13120467-002		Χ	Х				
	121913031	13120467-001		Χ	Х				
GroundWater	121713001	4090191001	180-28205-1			Χ	Χ	Χ	Х
	121713002	4090191002	180-28205-2			Χ	Х	Χ	Х
	121713003	4090191003	180-28205-3			Χ	Χ	Χ	Х
	121713004	4090191004	180-28205-4			Χ	Х	Χ	Х
	121713005	4090191005					Χ		
	121813006	4090245001	180-28280-1			Χ	Х	Χ	Х
	121813009	4090245008	180-28276-1			Χ	Χ	Χ	Х
	121813010	4090245006	180-28279-1			Χ	Х	Χ	Х
	121813012	4090245002	180-28280-2			Χ	Χ	Χ	Х
	121813013	4090245003	180-28280-3			Х	Х	Х	Х
	121813015	4090245007	180-28279-2			Χ	Χ	Χ	Х
	121813017	4090245004	180-28280-4			Х	Х	Х	Х
	121813018	4090245005					Χ		
	121913020	4090328001	180-28336-1			Х	Х	Х	Х
	121913023	4090328002	180-28336-2			Χ	Χ	Χ	Х
	121913027	4090326001	180-28338-1			Х	Х	Х	Х
	121913029	4090326002	180-28338-2			Χ	Χ	Χ	Х
	121913032	4090325001	180-28340-1			Х	Х	Х	Х
	121913033	4090328003					Χ		
	122013034	4090400001	180-28381-1			Х	Х	Х	Х
	122013035	4090400002	180-28381-2			Χ	Χ	Χ	Х

122013036	4090400003	180-28380-1	Х	Х	Х	Х
122013037	4090400004	180-28380-2	X	Χ	Х	Х
122013038	4090400005			Х		

2.0 INORGANIC DATA REVIEW

2.1 Summary

Blank, spiked, and duplicate results were provided. Overall, QC data indicated acceptable precision and accuracy. The results of the QC review are presented below.

2.2 Sample Receipt and Methodology

The aqueous samples were analyzed for inorganic parameters following the methods cited in the table 2-1.

Table 2-1. Water Inorganic Analytes and Methods Summary

Analytical Method	Analytes
EPA 6020	Metals
EPA OIA-1677	Available Cyanide

Generally, the samples arrived at the laboratories properly preserved and in good condition. A few of the available cyanide SDGs were noted for a Saturday delivery but did not arrive until Monday Dec 23. However, no data are qualified based upon this since sample receipt temperatures were still within the required specifications for thermal preservation. All samples were analyzed within the prescribed holding times where holding times have been defined.

2.3 Calibration

Initial instrument calibrations for each of the methods were all within acceptance criteria.

All of the initial calibration verification checks (ICVs) for these analyses met the \pm 10% acceptance criterion used by the laboratory and required by the methods. No data are qualified as a consequence of the initial calibration verification data.

The laboratory also performed the requisite interference checks (ICS A, ICS AB) with each calibration. All of the interference checks gave acceptable

results. Hence, no data are qualified as a consequence of the interference check sample data.

Continuing calibration verification checks were performed at the required frequencies. All of the continuing calibration verification checks (CCVs) for these analyses met the \pm 10% acceptance criterion used by the laboratory and required by the methods. No data are qualified as a consequence of the continuing calibration data.

2.4 Blanks

For SDG 4090191 initial and continuing calibration blanks (ICBs/CCBs) for antimony and lead gave values slightly above the limit of detection but below the reporting limit. The effected sample results were detected at greater than ten times the level of contamination, therefore no samples will be qualified based on this.

The initial and continuing calibration blanks (ICBs/CCBs) for available cyanide all gave results below the limit of detection. Therefore no data are qualified as a consequence of the calibration blank data.

Method blanks were prepared for each batch of samples prepared for analysis.

One batch had copper in the method blank. For SDG 4090191 copper gave a value slightly above the limit of detection but below the reporting limit. The effected sample result was detected at greater than ten times the level of contamination; therefore no samples will be qualified based on this.

The method blank for available cyanide all gave results below the limit of detection. Therefore, no data are qualified as a consequence of the method blank data.

The method blank results are summarized in Table 2-2 and 2-3.

Table 2-2. Water Method 6020 Method Blank Results Summary

Test Batch	Analyte	Units	Result
	Antimony	μg/L	1.0 U
150400	Beryllium	μg/L	1.0 U
150406	Copper	μg/L	0.27 J
	Lead	μg/L	1.0 U
150917	Lead	μg/L	1.0 U

Table 2-3. Water EPA Method OIA-1667 Method Blank Results Summary

Test Batch	Analyte	Units	Result
180-93124	Available cyanide	mg/L	0.0020 U
180-93210	Available cyanide	mg/L	0.0020 U

2.5 Laboratory Control Samples

Laboratory control samples (LCS) were analyzed with each of the data sets.

Laboratory control samples were prepared using commercially available reference materials.

The recovery limits used by the laboratory for LCS results are either those given in the method guidance or are based upon laboratory performance. No results exceeded these criteria; therefore, there is no need to qualify any results based on the LCS results.

Recoveries are given along with the acceptance limits in Tables 2-4 and 2-5.

Table 2-4. Water Method 6020 LCS Results Summary

QC Batch	Analyte	Recovery Limits Analyte (%)		Spike	Result	Recovery	
		Lower	Upper	(μg/L)	(μg/L)		
	Antimony	80	120	500	514	103	
150406	Beryllium	80	120	500	508	102	
150406	Copper	80	120	500	499	100	
	Lead	80	120	500	515	103	
150917	Lead	80	120	500	508	102	

Table 2-5. Water EPA Method OIA-1667 LCS Results Summary

QC Batch for	Analuta	Limits (%)	Spike	Result	Pagayam,	
OIA-1677	Analyte	Lower	Upper	(mg/L)	(mg/L)	Recovery
180-93124	Available cyanide	82	132	0.100	0.101	101
180-93210	Available cyanide	82	132	0.100	0.0984	98
160-93210	Available cyanide	82	132	0.100	0.102	102

2.6 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses were evaluated for each of the parameters at appropriate frequencies. On several occasions, the laboratory used non-project specific sample as matrix spike samples to satisfy batch QC requirements. However, only project requested MS/MSD results are included in this report.

No recoveries for 6020 or Available Cyanide fell outside of the limits required by the analytical method. Therefore no data are qualified as a consequence of the MS/MSD data.

The MS/MSD data are given in Tables 2-6 and 2-7.

Table 2-6. Water Method 6020 MS/MSD Sample Recoveries

	MS Sample ID: 121713001			MSD Sample ID: 121713001				Lab	
Analyte	Spike (μg/L)	MS Result (μg/L)	Rec (%)	Spike (µg/L)	MSD Result (μg/L)	Rec (%)	RPD	Sample Result (μg/L)	Max RPD
Antimony	500	514	103	500	520	104	1	0.34J	20
Beryllium	500	488	98	500	489	98	0	0.15 U	20
Copper	500	459	92	500	462	92	1	0.70 J	20
Lead	500	520	104	500	527	105	1	0.27J	20

Table 2-6. Water Method 6020 MS/MSD Sample Recoveries Cont

	MS Sample ID: 121813015			MSD Sample ID: 121813015				Lab	
Analyte	Spike (μg/L)	MS Result (μg/L)	Rec (%)	Spike (μg/L)	MSD Result (μg/L)	Rec (%)	RPD	Sample Result (µg/L)	Max RPD
Lead	500	509	102	500	503	101	1	0.13 J	20

Table 2-7. Water EPA Method OIA-1667 MS/MSD Sample Recoveries

		Sample II 21813015			MSD Sample ID: 121813015			Lab	
Analyte	Spike (mg/L)	MS Result (mg/L)	Rec (%)	Spike (mg/L)	MSD Result (mg/L)	Rec (%)	RPD	Sample Result (mg/L)	Max RPD
Available cyanide	0.100	0.0987	99	0.100	0.101	100	2	0.0020 U	20

2.7 Internal Standards

The National Functional Guidelines for Inorganic Data Review, October 2004 requires the relative intensity (%RI) for ICP/MS internal standards to fall within 60-125% for each sample analysis relative to the calibration standards. All internal standards met this requirement so no data will be qualified based on internal standards for ICP/MS.

2.8 ICP/MS Serial Dilutions

Serial dilution tests were performed by the laboratory on an analytical batch basis. However, only one project specific sample from this data set was subject to the serial dilution test.

All serial dilution tests met the acceptance criterion defined in the test method for all of the metals. Consequently no results are qualified due to serial dilution failures.

2.9 Field Duplicates

Field duplicates were collected and analyzed for the inorganic parameters. Field duplicates generally show excellent agreement for all of the analytes where the values are above five times the sample quantitation limit. Precision is only calculated where both the sample and the duplicate sample gave a positive result. Duplicate "NDs", however, are reported with 0% RPDs.

Criteria for evaluating field duplicate precision is provided in the Multi-Site QAPP Addendum dated March 12, 2012. Worksheet #28 of that addendum defines an upper limit, for values greater than five times the quantitation limit, at 30% RPD for precision between field duplicate values for inorganic parameters.

None of the field duplicates gave RPD values exceeding the 30% RPD limit specified in the QAPP Addendum. Therefore no data are qualified as a consequence of the duplicate data.

The results of the duplicate analyses are given in Tables 2-8 and 2-9.

Table 2-8. Method 6020 Field Dup Recoveries

		mple II 181301		Sample ID: 121813013			Sample ID: 122013034		Sample ID: 122013035					
Analyte	Result (μg/L)	Lab Flag	LOQ	Result (μg/L)	Lab Flag	LOQ	RPD	Result (μg/L)	Lab Flag	LOQ	Result (μg/L)	Lab Flag	LOQ	RPD
Lead	0.14	J	1.0	1.3		1.0	161.1	0.11	J	1.0	1	U	1.0	NC

Table 2-9. Water EPA Method OIA-1667 Field Dup Recoveries

	Sample	ID: 121	813012	Sample			
Analyte	Result Lab (mg/L) Flag		LOQ	Result (mg/L)	Lab Flag	LOQ	RPD
Available cyanide	0.019		0.0020	0.021		0.0020	10.0

Table 2-9. Water EPA Method OIA-1667 Field Dup Recoveries Cont.

	Sample	ID: 122	013034	Sample			
Analyte	Result (mg/L)	IN		Result (mg/L)	Lab Flag	LOQ	RPD
Available cyanide	0.0020	U	0.0020	0.0020	U	0.0020	0.0

3.0 ORGANIC DATA REVIEW

Blank, spiked, and duplicate results were provided. The results of the QC review are presented below. One method blank was prepared and analyzed with each analytical batch of samples.

Aqueous samples were analyzed for organic compounds following SW-846 Methods as shown in Table 3-1

Table 3-1. Organic Analytes and Methods Summary

Analytical Method	Analyte
EPA 8260B	Purgeable Volatile Organic Compounds (PVOC)
EPA 8270 by SIM	Polycyclic Aromatic Hydrocarbons (PAH)

3.1 SW-846 Method 8260B – Purgeable Volatile Organic Compounds

3.1.1 Summary

SW-846 Method 8260B employs gas chromatographic separation with a mass spectrometer as a detector.

3.1.2 Trip Blanks

Four trip blanks were provided with this sample set. None of the trip blanks associated with these samples gave results above the detection limit.

No data are qualified as a consequence of any of the field quality control blanks.

3.1.3 Method Blanks

The aqueous samples were analyzed in multiple analytical batches. The method blanks, showed no contamination above the detection limit. Hence, no data are qualified as a consequence of the method blank data.

The method blank data are summarized in Table 3-2.

Table 3-2. Water Method 8260B Method Blank Results Summary

Test Batch	Analyte	Units	Result
	1,2,4-Trimethylbenzene	μg/L	1.0 U
	1,3,5-Trimethylbenzene	μg/L	1.0 U
150514	Benzene	μg/L	1.0 U
150514	Ethylbenzene	μg/L	1.0 U
	Toluene	μg/L	1.0 U
	Xylene (Total)	μg/L	1.3 U
	1,2,4-Trimethylbenzene	μg/L	1.0 U
	1,3,5-Trimethylbenzene	μg/L	1.0 U
150684	Benzene	μg/L	1.0 U
150084	Ethylbenzene	μg/L	1.0 U
	Toluene	μg/L	1.0 U
	Xylene (Total)	μg/L	1.3 U
	1,2,4-Trimethylbenzene	μg/L	1.0 U
	1,3,5-Trimethylbenzene	μg/L	1.0 U
150057	Benzene	μg/L	1.0 U
150857	Ethylbenzene	μg/L	1.0 U
	Toluene	μg/L	1.0 U
	Xylene (Total)	μg/L	1.3 U

3.1.4 Calibration

All initial calibration criteria were met for all compounds. All analytes fit first order linear regression curves and gave average response factors (RFs) with <15% RSD over the average. Therefore average RFs were used in sample quantitation. No data are qualified as a result of the initial calibration data.

For evaluating calibration verifications, the June 2008 CLP National Functional Guidelines have established a \pm 40% drift or difference acceptability criterion for analytes known to exhibit poor response and a \pm 25% drift or difference criterion for all other target analytes. None of the analytes of concern in this investigation are considered to exhibit poor response. The calibration verification associated with this data set did not exceed the \pm 25% difference

criterion in place for all other target analytes. Consequently, no data are qualified as a result of the calibration verification data.

3.1.5 Internal Standard Areas

No sample analyses reported in this data set have internal standard areas less than -50% or greater than +100% of the area response of the corresponding continuing calibration verification. Therefore, no data are qualified.

3.1.6 Surrogate Compound Recoveries

Three surrogate compounds, 4-bromofluorobenzene, toluene- d_8 , and dibromofluoromethane, were spiked into each field sample to monitor analyte recovery in the analytical system. The surrogates used by the laboratory are acceptable to measure recovery under EPA SW-846 guidance for this analytical method.

Recoveries for all surrogates for all samples were well within the acceptance limits. No data require qualification based upon surrogate recoveries.

Recoveries for all surrogates for all samples are presented in Table 3-3.

Table 3-3. Water Method 8260B Surrogate Recoveries

Lab Sample Number	Field ID	Dilution	4 Bromofluo		Dibromofluo	promethane	Toluene- d ₈	
		Limits:	43	137	70	130	55	137
4090191001	121713001	1	79		98		91	
4090191002	121713002	1	75		97		92	
4090191003	121713003	1	74		98		93	
4090191004	121713004	1	70		103		92	
4090191005	121713005	1	70		104		91	
4090245001	121813006	1	98		101		104	
4090245002	121813012	1	96		101		103	
4090245003	121813013	1	97		101		103	
4090245004	121813017	1	97		101		103	
4090245005	121813018	1	98		102		104	
4090245006	121813010	4	101		101		104	
4090245007	121813015	1	98		100		104	
4090245008	121813009	1	97		101		104	
4090325001	121913032	1	97		101		104	
4090326001	121913027	1	97		102		105	
4090326002	121913029	1	99		102		104	
4090328001	121913020	1	97		100		104	
4090328002	121913023	1	98		102		104	
4090328003	121913033	1	98		101		103	
4090400001	122013034	1	97		99		103	
4090400002	122013035	1	97		101		103	
4090400003	122013036	1	98		100		104	
4090400004	122013037	1	97		102		104	
4090400005	122013038	1	97		100		103	

3.1.7 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses were performed on one sample as specified by the project team in accordance with the Sampling and Analysis Plan. None of the target compounds recovered outside of the limits established by the laboratory. The spike solution used by the laboratory does not contain 1,2,4-Trimethylbenzene or 1,3,5-Trimethylbenzene. Therefore there are no spike results for the LCS or MS/MSD for these analytes.

No action is defined for flagging data based on the MS/MSD results or RPD values alone. Since all of the reported recoveries were within acceptance limits, no data are qualified as a result of the matrix spike/matrix spike duplicate analyses.

The MS/MSD results are summarized in Table 3-4.

Table 3-4. Water Method 8260B MS/MSD Sample Recoveries

	MS Sample ID: 121813015				D Sample 12181301			Lab		
Analyte	Spike (µg/L)	MS Result (μg/L)	Rec (%)	Spike (μg/L)	MSD Result (μg/L)	Rec (%)	RPD	Sample Result (μg/L)	Max RPD	
Benzene	50	57.1	114	50	56	112	2	1.0 U	20	
Ethylbenzene	50	54.6	109	50	53	106	3	1.0 U	20	
Toluene	50	53.9	108	50	52.7	105	2	1.0 U	20	
Xylene (Total)	150	161	107	150	158	105	2	1.3 U	20	

3.1.8 Laboratory Control Samples

A Laboratory Control Sample (LCS) analysis was performed for each batch of samples analyzed. None of the analytes recovered outside of the acceptance limits established by the laboratory. No data are qualified due to failed LCS recoveries. The spike solution used by the laboratory does not contain 1,2,4-Trimethylbenzene or 1,3,5-Trimethylbenzene. Therefore there are no spike results for the LCS or MS/MSD for these analytes.

The LCS results are summarized in Table 3-5.

Table 3-5. Water Method 8260B LCS Summary

OC Batch	Anglista	Recovery	Limits (%)	Spike	Result	Basayanı
QC Batch	Analyte	Lower	Upper	(μg/L)	(μg/L)	Recovery
	Benzene	70	137	50	52.9	106
450544	Ethylbenzene	70	130	50	51.9	104
150514	Toluene	70	130	50	54.0	108
	Xylene (Total)	70	130	150	177	118
	Benzene	70	137	50	57.0	114
150684	Ethylbenzene	70	130	50	55.0	110
150084	Toluene	70	130	50	53.9	108
	Xylene (Total)	70	130	150	162	108
	Benzene	70	137	50	55.4	111
150057	Ethylbenzene	70	130	50	53.6	107
150857	Toluene	70	130	50	52.9	106
	Xylene (Total)	70	130	150	160	106

3.1.9 Field Duplicates

Field duplicates generally have good agreement for all of analytes with all RPD values <30%. Precision is only calculated where both the sample and the duplicate sample gave a positive result. Duplicate "NDs", however, are reported with 0% RPDs. No results will be qualified based on field duplicate data for 8260.

The results of the field duplicate analyses are given in Table 3-6.

Table 3-6. Water Method 8260B Field Dup Results

		nple II 181301		Sai 121			
Analyte	Result (μg/L)	Lab Flag	LOQ	Result (μg/L)	Lab Flag	LOQ	RPD
1,2,4-Trimethylbenzene	1.0	U	1.0	1.0	U	1.0	0.0
1,3,5-Trimethylbenzene	1.0	U	1.0	1.0	U	1.0	0.0
Benzene	1.0	U	1.0	1.0	U	1.0	0.0
Ethylbenzene	1.0	U	1.0	1.0	U	1.0	0.0
Toluene	1.0	U	1.0	1.0	U	1.0	0.0
Xylene (Total)	3.0	U	3.0	3.0	U	3.0	0.0

Table 3-6. Water Method 8260B Field Dup Results Cont

		nple II 201303		Sar 122			
Analyte	Result (μg/L)	Lab Flag	LOQ	Result (μg/L)	Lab Flag	LOQ	RPD
1,2,4-Trimethylbenzene	1.0	U	1.0	1.0	U	1.0	0.0
1,3,5-Trimethylbenzene	1.0	U	1.0	1.0	U	1.0	0.0
Benzene	1.0	U	1.0	1.0	U	1.0	0.0
Ethylbenzene	1.0	U	1.0	1.0	U	1.0	0.0
Toluene	1.0	U	1.0	1.0	U	1.0	0.0
Xylene (Total)	3.0	U	3.0	3.0	U	3.0	0.0

3.2 SW-846 Method 8270C/SIM-PAHs

3.2.1 Summary

SW-846 Method 8270C/SIM employs gas chromatographic separation with mass spectroscopic identification using selected ion monitoring (SIM).

3.2.2 Method Blanks

The method blanks, showed contamination above the detection limit but less than the reporting limit for naphthalene and 2-Methylnaphthalene. The effected sample results that are above the detection limit but below the reporting limit will be reported at the reporting limit and qualified with a "U" (not detected). Results that are above the reporting limit, but less than five times the reporting limit, will be qualified as estimated "J".

The results for the method blanks are summarized in Table 3-7.

Table 3-7. Water Method 8270-SIM Method Blank Results Summary

Analyte	Units	QC Batch: 150730	QC Batch: 150988
2-Methylnaphthalene	μg/L	0.0049 J	
Acenaphthene	μg/L	0.050 U	
Acenaphthylene	μg/L	0.050 U	
Anthracene	μg/L	0.050 U	
Benzo(a)anthracene	μg/L	0.050 U	
Benzo(a)pyrene	μg/L	0.050 U	
Benzo(b)fluoranthene	μg/L	0.050 U	
Benzo(g,h,i)perylene	μg/L	0.050 U	
Benzo(k)fluoranthene	μg/L	0.050 U	
Chrysene	μg/L	0.050 U	
Dibenz(a,h)anthracene	μg/L	0.050 U	
Fluoranthene	μg/L	0.050 U	
Fluorene	μg/L	0.050 U	
Indeno(1,2,3-cd)pyrene	μg/L	0.050 U	
Naphthalene	μg/L	0.010 J	0.0065 J
Phenanthrene	μg/L	0.050 U	
Pyrene	μg/L	0.050 U	

3.2.3 Calibration

Instrument tuning checks using decafluorotriphenylphosphine (DFTPP) were performed daily and every 12 hours as described in the methods. However, since this method employs selected ion monitoring, tuning using DFTPP has little value. Consequently, no data are qualified based upon DFTPP tuning criteria.

The initial instrument calibration performed for this method gave satisfactory results with response factors over the calibration range <15% RSD. Therefore an average response factor calibration model was used to quantitate all compounds results.

The initial calibration verifications (ICV) reported with this data set gave percent differences less than the 25% limit defined in the National Functional Guidelines for calibration verification. Therefore, no results are qualified as a consequence of the initial calibration verifications.

All of the continuing calibration verification (CCV) checks for PAH analyses performed gave acceptable results (i.e., <25% D using the CLP National Functional Guidelines) for all of the target analytes. No data are qualified as a consequence of the continuing calibration data.

The peak shapes and chromatographic resolution for the isomers benzo(b)fluoranthene and benzo(k)fluoranthene evident in the sample chromatograms for the samples indicate that the two isomers are not adequately resolved to be quantitated separately as the laboratory attempted to do. The laboratory's report narratives noted this issue but stopped short of reporting the two isomers as a coeluting pair (as is done for m/p-xylene). Consequently all positive results for benzo(b)fluoranthene and benzo(k)fluoranthene in all samples for these two isomers are qualified as estimated ("J").

3.2.4 Internal Standard Areas

One sample analyses reported in this data set yielded internal standard areas less than 50% of the area response of the corresponding continuing calibration verification. In this case the only analyte reported was naphthalene, and the failure was perlene- d_{12} . This internal standard is not associated with naphthalene. Therefore, no data are qualified.

3.2.5 Surrogate Compound Recoveries

Two surrogates, 2-fluorobiphenyl, and terphenyl- d_{14} , were spiked into each field sample to monitor method recovery. Given the focused nature of the compounds of concern (i.e., PAHs), the surrogates reported should be adequate to monitor recovery in the analyses. Two samples had surrogates with 0% recovery due to sample dilution. Under these circumstances qualification of data is not warranted. No data is qualified due to surrogate recoveries.

The surrogate recoveries for all samples are presented in Table 3-8.

Table 3-8. Water Method 8270-SIM Surrogate Recoveries

Lab Sample Number	Field ID	Dilution	2- Fluorobiphenyl		Terphenyl-d ₁₄		
		Limits:	24	130	44	169	
4090191001	121713001	1	58		98		
4090191002	121713002	1	65		99		
4090191003	121713003	1	47		92		
4090191004	121713004	1	53		93		
4090245001	121813006	1	57		102		
4090245002	121813012	1	49		105		
4090245003	121813013	1	50		98		
4090245004	121813017	1	49		125		
4090245006	121813010	100	0		0		
4090245007	121813015	1	50		88		
4090245008	121813009	1	50		104		
4090325001	121913032	1	50		97		
4090326001	121913027	1	41		79		
4090326002	121913029	50	0		0		
4090328001	121913020	1	50		83		
4090328002	121913023	1	46		92		
4090400001	122013034	1	46		80		
4090400002	122013035	1	51		91		
4090400003	122013036	1	49		86		
4090400004	122013037	1	54		98		

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3.2.6 Matrix Spike/Matrix Spike Duplicates

Sample 121813015 was used to perform MS/MSD analyses for 8270-SIM. Guidance in the National Functional Guidelines does not call for qualifying data based upon the matrix spike analyses alone. No data are qualified based upon the MS/MSD results.

The MS/MSD recoveries for all samples are presented in Table 3-9.

Table 3-9. Water Method 8270-SIM MS/MSD Sample Recoveries

		Sample 2181301			D Sample 2181301			Lab	
Analyte	Spike (μg/L)	MS Result (μg/L)	Rec (%)	Spike (μg/L)	MSD Result (μg/L)	Rec (%)	RPD	Sample Result (μg/L)	Max RPD
Naphthalene	.19	0.11	54	.19	0.11	54	3	0.0067 JB	50

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3.2.7 Laboratory Control Samples

A laboratory control sample (LCS) was prepared and analyzed with each batch of samples. All of the analytes for the laboratory control samples recovered within the limits used by the laboratory.

The laboratory control sample results are given in Table 3-10.

Table 3-10. Water Method 8270-SIM LCS Results Summary

	Coile	Rec Lin	nits (%)	QC Batch	: 150730	QC Batch:	150988
Analyte	Spike (Units)	Lower	Upper	Result (μg/L)	Rec (%)	Result (μg/L)	Rec (%)
2-Methylnaphthalene	.2	32	130	0.17	87		
Acenaphthene	.2	30	130	0.16	81		
Acenaphthylene	.2	28	130	0.16	81		
Anthracene	.2	22	130	0.16	82		
Benzo(a)anthracene	.2	40	130	0.18	89		
Benzo(a)pyrene	.2	51	130	0.18	88		
Benzo(b)fluoranthene	.2	45	130	0.18	91		
Benzo(g,h,i)perylene	.2	59	130	0.20	99		
Benzo(k)fluoranthene	.2	60	130	0.23	113		
Chrysene	.2	62	130	0.21	105		
Dibenz(a,h)anthracene	.2	51	130	0.16	78		
Fluoranthene	.2	43	130	0.17	85		
Fluorene	.2	29	130	0.17	85		
Indeno(1,2,3-cd)pyrene	.2	56	130	0.17	86		
Naphthalene	.2	30	130	0.17	86	0.17	87
Phenanthrene	.2	29	130	0.16	81		
Pyrene	.2	38	130	0.19	97		

3.2.8 Field Duplicates

Field duplicates generally show good agreement for all of the analytes. Precision is only calculated where both the sample and the duplicate sample gave a positive result (NC=Not Calculated). Duplicate "NDs", however, are reported with 0% RPDs. The National Functional Guidelines do not provide any guidance for qualifying data associated with field or sample duplicates for semivolatiles analyses. However, requirements that appear in the Multi-Site QAPP Addendum governing this project place a 30% limit on the RPD values where the results are >2x the limit of quantitation. No results for any field samples associated with these duplicate pairs are qualified based upon field duplicate data.

The results of the duplicate analyses are given in Table 3-11.

Table 3-11. Water Method 8270-SIM Field Dup Results

Anglista		mple II 18130:			mple II 18130:		
Analyte	Result (μg/L)	Lab Flag	LOQ	Result (μg/L)	Lab Flag	LOQ	RPD
Naphthalene	0.016	JB	0.050	0.070	В	0.049	125.6

Table 3-11. Water Method 8270-SIM Field Dup Results Cont

Anglista		mple II 201303			mple I 20130:		222
Analyte	Result (μg/L)	Lab Flag	LOQ	Result (μg/L)	Lab Flag	LOQ	RPD
Naphthalene	0.0067	JB	0.054	0.005	U	0.049	NC

4.0 VAPOR SAMPLE ANALYSES

Soil vapor phase samples were collected as part of this investigation. Blank, laboratory control sample, and duplicate results were provided. The results of the QC review are presented below.

Vapor phase samples were analyzed for organic compounds following the methods as shown in Table 4-1.

Table 4-1. Vapor Phase Analytes and Methods Summary

Analytical Method	Analyte			
EPA Method TO-15	Volatile Organic Compounds (VOCs)			
ASTM D1946/EPA Method 3C	Oxygen, Carbon Dioxide, Methane			

All samples were collected in SUMMA polished canisters and received by the laboratory in good condition and intact. No data are qualified based upon sample receipt conditions.

All sample analyses were performed within the EPA-established holding times. No data are qualified based upon sample holding times.

STAT laboratory noted on their case narrative that the methylene chloride reported in sample 121913031 is a possible lab artifact. Based on this the sample will be qualified as unusable "R" for methylene chloride.

4.1 EPA Method TO-15: Volatile Organic Compounds (VOCs)

4.1.1 Summary

EPA Method TO-15 employs gas chromatographic separation with a mass spectrometer as a detector. One method blank was prepared and analyzed with each analytical batch of samples. Ultra High Purity nitrogen was used as the matrix for VOC method blank analysis.

4.1.2 Method Blanks

The samples were analyzed in two analytical batches. Methylene chloride and napthalene were detected in the method blanks above the limit of detection but below the reporting limit. All positive results for methylene chloride and naphthalene in the associated batches will be qualified as estimated "J".

The results for the method blanks are summarized in Table 4-2.

Table 4-2. EPA TO-15 Method Blank Summary

Analyte	Units	QC Batch: R95580	QC Batch: R95604
1,1,1-Trichloroethane	μg/m³	1.1 U	1.1 U
1,1-Dichloroethane	μg/m³	0.8 U	0.8 U
1,2,4-Trimethylbenzene	μg/m³	1 U	1 U
2-Butanone	μg/m³	1.5 U	1.5 U
Acetone	μg/m³	4.8 U	4.8 U
Benzene	μg/m³	0.6 U	0.6 U
Carbon disulfide	μg/m³	0.62 U	0.62 U
cis-1,2-Dichloroethene	μg/m³	0.8 U	0.8 U
Ethylbenzene	μg/m³	0.9 U	0.9 U
Methylene chloride	μg/m³	0.6947 J	0.59 U
Naphthalene	μg/m³	0.26 U	0.05242 J
Styrene	μg/m³	0.9 U	0.9 U
Tetrachloroethene	μg/m³	1.4 U	1.4 U
Toluene	μg/m³	0.8 U	0.8 U
trans-1,2-Dichloroethene	μg/m³	0.8 U	0.8 U
Vinyl chloride	μg/m³	0.5 U	0.5 U
Xylenes, Total	μg/m³	2.6 U	2.6 U

4.1.3 Calibration

The initial instrument calibration performed for this method gave satisfactory results with response factors over the calibration range <30% RSD. Therefore an average response factor calibration model was used to quantitate all target analyte results.

All of the initial calibration verification (ICV) and continuing calibration verification (CCV) checks for Method TO-15 performed gave acceptable results (i.e., <30%D) for all of the target analytes.

No data are qualified as a consequence of the calibration data.

4.1.4 Surrogate Compound Recoveries

Surrogate Compound analysis is not included as part of EPA Method TO-15.

4.1.5 Laboratory Control Samples

A laboratory control sample (LCS) was prepared and analyzed with each batch of samples.

All of the target analytes for all of the laboratory control samples recovered within the limits used by the laboratory. No data are qualified due to failed LCS recoveries.

The laboratory control sample results are given in Table 4-3.

Table 4-3. EPA TO-15 Laboratory Control Sample Summary

	6.31	Rec Lin	nits (%)	QC Batcl	h: R95580	QC Batcl	h: R95604
Analyte	Spike (μg/m³)	Lower	Upper	Result (μg/m³)	Recovery (%)	Result (μg/m³)	Recovery (%)
1,1,1-Trichloroethane	27.28	70	130	27.23	99.8	26.63	97.6
1,1-Dichloroethane	20.24	70	130	21.05	104	18.78	92.8
1,2,4-Trimethylbenzene	24.58	70	130	25.56	104	25.61	104
2-Butanone	14.75	70	130	15.69	106	14.6	99
Acetone	11.88	70	130	12.33	104	11.05	93
Benzene	15.97	70	130	13.96	87.4	13.99	87.6
Carbon disulfide	15.57	70	130	16.32	105	14.29	91.8
cis-1,2-Dichloroethene	19.82	70	130	20.97	106	18.99	95.8
Ethylbenzene	21.71	70	130	20.8	95.8	20.75	95.6
Methylene chloride	17.37	70	130	18.41	102	16.22	93.4
Naphthalene	26.21	70	130	30.82	118	31.92	122
Styrene	21.3	70	130	22.58	106	22.83	107
Tetrachloroethene	33.91	70	130	34.86	103	33.44	98.6
Toluene	18.84	70	130	18.46	98	18.28	97
trans-1,2-Dichloroethene	19.82	70	130	21.69	109	19.15	96.6
Vinyl chloride	12.78	70	130	13.5	106	11.66	91.2
Xylenes, Total	65.13	70	130	65.22	100	65.82	101

4.1.6 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses are not performed for EPA Method TO-15 analyses.

4.1.7 Field Duplicates

Field duplicates generally show good agreement with RPD <30% for all nalytes. Precision is only calculated where both the sample and the duplicate sample gave a positive result (NC=Not Calculated). Duplicate "NDs", however, are reported with 0% RPDs.

The National Functional Guidelines do not provide any guidance for qualifying data associated with field or sample duplicates for semivolatiles analyses. However, requirements that appear in the Multi-Site QAPP Addendum governing this project place a 30% limit on the RPD values where the results are >2x the limit of quantitation. No results will be qualified based on field duplicate data.

The results of the duplicate analyses are given in Table 4-4.

Table 4-4. EPA TO-15 Field Dup Sample Summary

		ple ID 91302			ple ID 91302		
Analyte	Result (μg/m³)	Lab Flag	LOQ	Result (μg/m³)	Lab Flag	LOQ	RPD
1,1,1-Trichloroethane	2	U	2	2	U	2	0.0
1,1-Dichloroethane	1.5	U	1.5	1.5	U	1.5	0.0
1,2,4-Trimethylbenzene	1.9	U	1.9	1.8	U	1.8	0.0
2-Butanone	2.8	U	2.8	2.7	U	2.7	0.0
Acetone	8.9	U	8.9	8.7	U	8.7	0.0
Benzene	1.1	U	1.1	1.1	U	1.1	0.0
Carbon disulfide	1.2	U	1.2	1.1	U	1.1	0.0
cis-1,2-Dichloroethene	1.5	U	1.5	1.5	U	1.5	0.0
Ethylbenzene	1.7	U	1.7	1.6	U	1.6	0.0
Methylene chloride	13	U	13	13	U	13	0.0
Naphthalene	0.48	U	0.48	0.47	U	0.47	0.0
Styrene	1.7	U	1.7	1.6	U	1.6	0.0
Tetrachloroethene	2.6	U	2.6	2.6	U	2.6	0.0
Toluene	1.5	U	1.5	1.5	U	1.5	0.0
trans-1,2-Dichloroethene	1.5	U	1.5	1.5	U	1.5	0.0
Vinyl chloride	0.93	U	0.93	0.91	U	0.91	0.0
Xylenes, Total	4.8	U	4.8	4.7	U	4.7	0.0

Table 4-4. EPA TO-15 Field Dup Sample Summary Cont

		ple ID 913030			ple ID 91303:		
Analyte	Result (μg/m³)	Lab Flag	LOQ	Result (μg/m³)	Lab Flag	LOQ	RPD
1,1,1-Trichloroethane	2	U	2	1.9	U	1.9	0.0
1,1-Dichloroethane	1.5	U	1.5	1.4	U	1.4	0.0
1,2,4-Trimethylbenzene	1.8	U	1.8	1.8	U	1.8	0.0
2-Butanone	2.7	U	2.7	2.6	U	2.6	0.0
Acetone	8.8	U	8.8	8.5	U	8.5	0.0
Benzene	1.1	U	1.1	1.1	U	1.1	0.0
Carbon disulfide	1.1	U	1.1	1.1	U	1.1	0.0
cis-1,2-Dichloroethene	1.5	U	1.5	1.4	U	1.4	0.0
Ethylbenzene	1.6	U	1.6	1.6	U	1.6	0.0
Methylene chloride	13	U	13	28		12	NC
Naphthalene	0.48	U	0.48	0.46	U	0.46	0.0
Styrene	1.6	U	1.6	1.6	U	1.6	0.0
Tetrachloroethene	2.6	U	2.6	2.5	U	2.5	0.0
Toluene	1.5	U	1.5	1.4	U	1.4	0.0
trans-1,2-Dichloroethene	1.5	U	1.5	1.4	U	1.4	0.0
Vinyl chloride	0.91	U	0.91	0.88	U	0.88	0.0
Xylenes, Total	4.8	U	4.8	4.6	U	4.6	0.0

4.2 EPA Method 3C: Oxygen, Carbon Dioxide, and Methane

4.2.1 Summary

EPA Method 3C employs gas chromatographic separation with thermal conductivity detector.

4.2.2 Method Blanks

The samples were analyzed in several analytical batches. None of the target compounds were detected in the method blanks.

No data are qualified due to the blank contamination.

The results for the method blanks are summarized in Table 4-5.

Table 4-5. EPA Method 3C Method Blank Summary

Parameter	Batch	Units	Result
Carbon Dioxide	R95544	mol %	0.08 U
	R95570	mol %	0.08 U
N.4 a t la a a	R95544	mol %	0.1 U
Methane	R95570	mol %	0.1 U
	R95544	mol %	0.8 U
Oxygen	R95570	mol %	0.8 U

4.2.3 Calibration

The initial instrument calibration performed for this method gave satisfactory results with response factors over the calibration range <10% RSD. Therefore an average response factor calibration model was used to quantitate all target analyte results.

All of the initial calibration verification (ICV) and continuing calibration verification (CCV) checks for Method 3C performed gave acceptable results (i.e., <10%D) for all of the target analytes.

No data are qualified as a consequence of the calibration data.

4.2.4 Surrogate Compound Recoveries

Surrogate Compound analysis is not included as part of EPA Method 3C.

4.2.5 Laboratory Control Samples

A laboratory control sample (LCS) was prepared and analyzed with each batch of samples.

All of the target analytes for each of the laboratory control samples recovered within the limits used by the laboratory. Based upon the acceptable recoveries, there is no need to qualify data based upon the LCS recovery results.

The laboratory control sample results are given in Table 4-6.

Table 4-6. EPA Method 3C Laboratory Control Sample Summary

	Cailea	Rec Lin	nits (%)	QC Batch	h: R95544	QC Batch: R95570		
Analyte	Spike (mol %)	Lower	r Upper	Result (mol %)	Recovery (%)	Result (mol %)	Recovery (%)	
Carbon Dioxide	0.6	80	120	0.676	113	0.654	109	
Methane	1	80	120	0.992	99.2	0.988	98.8	
Oxygen	0.8	80	120	0.806	101	0.812	102	

4.2.6 Matrix Spike/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) analyses are not performed for EPA Method 3C analyses.

4.2.7 Field Duplicates

Field duplicates show excellent agreement with RPD <30% for all the analytes. Precision is only calculated where both the sample and the duplicate sample gave a positive result (NC=Not Calculated). Duplicate "NDs", however, are reported with 0% RPDs.

Based upon these observations, no results for any field samples associated with these duplicate pairs are qualified based upon field duplicate data.

The results of the duplicate analyses are given in Table 4-7.

Table 4-7. EPA Method 3C Field Dup Sample Summary

Analyta		ple ID 913024			ple ID: 13025		
Analyte	Result (mol %)	Lab Flag		Result (mol %)	Lab Flag	LOQ	RPD
Carbon Dioxide	0.402		0.08	0.408		0.08	1.5
Methane	0.1	U	0.1	0.1	U	0.1	0.0
Oxygen	17		0.8	17.2		0.8	1.2

Table 4-7. EPA Method 3C Field Dup Sample Summary Cont

Analyte	Sample ID: 121913030			Sample ID: 121913031			222
	Result (mol %)	Lab Flag	LOQ	Result (mol %)	Lab Flag	LOQ	RPD
Carbon Dioxide	0.096		0.08	0.098		0.08	2.1
Methane	0.1	U	0.1	0.1	U	0.1	0.0
Oxygen	17.6		0.8	17.6		0.8	0.0